AN ASSESSMENT OF VARIABILITY IN THE AVERAGE TENSILE PROPERTIES OF A MELT-INFILTRATED SiC/SiC COMPOSITE

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ABSTRACT

Woven, SiC/SiC Ceramic Matrix Composites (CMCs), manufactured by the slurry-cast, melt-infiltration process are under consideration as combustor liner materials in aircraft gas turbine engines. Tensile properties (elastic modulus, proportional limit strength, in-plane tensile strength, and strain to failure) of the CMC, manufactured during two separate time periods (9/99 and 1/01), were determined at 816 and 1204°C by conducting tensile tests on specimens machined from the CMC plates. A total of 24 tensile tests were conducted for each temperature and CMC variant combination. In this study, average tensile properties of the two CMC variants were statistically compared to evaluate significant differences, if any, within the CMC's properties. A random sampling method was also used to assess the variability exhibited by the mean values of the four tensile properties. By varying the group size of the random sample, the minimum number of tensile tests required to characterize the mean value of each tensile property for the CMC was determined.

INTRODUCTION

Materials capable of withstanding high temperatures, particularly Ceramic Matrix Composites (CMCs), are identified for application in the combustor and nozzle components in the next generation aircraft propulsion systems [1]. Design of propulsion system components necessitates the development of reliable mechanical property data bases for the CMCs. Although the manufacturing process and specifications should be consistent and robust enough to yield a material with minimum variations in mechanical properties, a reliable estimate of mechanical properties has overriding importance to a design. In this paper the relationship between the number of repeated tests and the variation in the average tensile properties of two variants of a silicon carbide based CMC is determined at two temperatures. The two material systems studied were the 9/99 and 1/01 CVI SiC/slurry-cast/melt-infiltration composites manufactured by General Electric Power Systems Composites, LLC. In order to incorporate lot-to-lot statistical variations, an even sampling of test specimens was selected from three separate manufacturing lots for both variants of the CMC.

Average values of the tensile properties from both variants of the CMC were compared with a Student's t-test to evaluate the similarity of each property. The minimum number of tests required for obtaining the average value of a tensile property to within a certain percent error was

determined with a simulation that involved random sampling of the data from both variants of the CMC. Results obtained from the random sampling study were used to provide guidelines for characterizing the tensile properties of similar classes of CMCs.

EXPERIMENTAL DETAILS

The fiber pre-form for both the 9/99 and 1/01 MI SiC/SiC composites consisted of Sylramic™ fiber woven to a 5HS weave, 20 EPI configuration with a $[0/90]_{4S}$ lay-up. For the 1/01 material, fiber pre-forms were iBN heat treated prior to matrix densification. Both materials had a CVI silicon-doped BN interphase and a CVI SiC matrix fully densified by the slurry-cast, melt infiltration process. The manufacturing process yielded plates that were 229, 152, and 2 mm in length, width, and thickness, respectively. Tensile specimens with a test section width and length of 10.2 mm and 28 mm, respectively, and an overall length of 152 mm were machined from the CMC plates. Geometry of the test specimen was reported in more detail previously [2]. For both the 9/99 and 1/01 variants of the CMC, a distribution of four tensile specimens per plate and two plates per lot for each of the three tested lots were selected to obtain a total population of 24 specimens. Additional details on test specimen grips, test frame alignment procedure, specimen heating, and strain measurement are described in Refs. [2-4].

TENSILE DATABASE

Tensile tests were conducted on both the 9/99 and 1/01 test specimens at 816°C and 1204°C. For each material and temperature combination, a total of 24 tensile tests were conducted. Tensile properties of the 9/99 material at 1204°C were previously reported [2]. However, tensile properties of the 9/99 material at 816 as well as 1204°C are reported in this paper for comparison with corresponding values from the 1/01 material. Mean values and standard deviations of four tensile properties (elastic modulus, E; proportional limit strength, PLS (0.005% offset); in-plane tensile strength, ITS; and strain to failure, SF) at both temperatures are shown in Tables I and II for the 9/99 and 1/01 materials, respectively. In these tables, for each tensile property the first number is the mean value and the second number is the standard deviation.

The mean values of E, PLS, and ITS in both the 9/99 and 1/01 materials decreased as the temperature increased from 816 to 1204°C. No such clear trend was exhibited by the SF. At both temperatures, no major differences were observed in the mean E values between the two variants of the CMC. The mean PLS values of the 1/01 material were marginally lower than the corresponding values of the 9/99 material at both the temperatures. However, mean values of ITS and SF for the 1/01 material were higher than those for the 9/99 material. In general, for all the four tensile properties at both temperatures, the standard deviations exhibited by the 9/99 material were lower than those for the 1/01 material.

Mean values of all the tensile properties of the 9/99 and 1/01 materials were compared with a two-tailed Student's t-test and a risk level, $\alpha = 0.02$. No statistically significant differences were found to exist among the mean values of E and PLS at both the temperatures between the two variants of the CMC. However, statistically significant differences were determined to exist among the mean values of ITS and SF between the two variants of the CMC. Results of these analyses indicate that for the MI SiC/SiC composite, even though differences exist in the mean values of the tensile properties from one variant to another, for some of the tensile properties (E and PLS), these differences are not statistically significant.

Table I. Means and Standard Deviations of Tensile Properties for 9/99 MI SiC/SiC Composite [n = 24]

Temperature [°C]	E [GPa]	PLS [MPa]	ITS [MPa]	SF [%]
816	208 {14}*	177 {19}	362 {32}	0.48 {0.06}
1204	182 {14}	166 {28}	307 {21}	0.46 {0.07}

* {} Denotes Standard Deviation

Table II. Means and Standard Deviations of Tensile Properties for 1/01 MI SiC/SiC Composite [n = 24]

Temperature [°C]	E [GPa]	PLS [MPa]	ITS [MPa]	SF [%]
816	201 {26}*	167 {25}	436 {32}	0.54 {0.09}
1204	184 {25}	155 {28}	399 {37}	0.57 {0.12}

* {} Denotes Standard Deviation

SIMULATION OF GROUPS BY RANDOM SAMPLING

The number of test specimens required to characterize the mean values of the mechanical properties of the MI SiC/SiC composite is important both to the experimentalist as well as the designer of propulsion system components. A large number of tests definitely increases the reliability of the mean values of the properties for the designer. However, they can be prohibitively expensive from an experimentalist's point of view. An attempt was made in this study to determine the mean values of the tensile properties of the composite to within predetermined error bands. This was accomplished by assuming that for both the 9/99 and 1/01 materials, the original sets of 24 data points at 816 and 1204°C represent the populations at the respective conditions. For each tensile property and test temperature, groups of 3 to 12 data points were randomly selected and the means and standard deviations associated with each group were calculated. The number of groups chosen for the simulation should be sufficiently large to capture a significant amount of the variation exhibited by the mean values of the tensile properties. Initially 30, 50, and 200 groups were used in the analyses and finally 200 groups were determined to be sufficient to estimate the variability in the mean values. Therefore, a total of 200 groups were randomly selected for each tensile property and duplication of samples (data points) within a group was avoided. In addition, permutations of the samples were not permitted within the 200 groups. Hence, the 200 groups essentially simulated independent combinations. The variability exhibited by the means of the randomly selected 200 groups with different sample set sizes ($k = 3$ to 12) was used to estimate the minimum number of samples required to estimate the mean values of the tensile properties to within 10 and 20% of the corresponding population mean values (Tables I and II).

RESULTS OF SIMULATION

For a given tensile property average values of the property from 200 groups were calculated. These mean values follow a normal distribution, regardless of the distribution followed by the

parent population [5]. An example of these distributions (for group sizes, k = 4, 8, and 12) is shown in Fig. 1 for E of the 9/99 material at 816°C. As expected, as the number of samples in the group increased from 4 to 12, the variation of the distributions decreased. Also, the mean values of all the three distributions approached the mean value of the parent population (Table I). For a given group size and tensile property, the maximum and minimum average values among the 200 randomly selected sample groups served as indicators of the bounds in variability.

For the 9/99 material the variability in the group averages of E are plotted with respect to the group size in Fig. 2. Only the maximum and minimum group average values are depicted with data points for simplicity. The line connecting these two points represents a distribution (similar to those shown in Fig. 1) and contains the remaining 198 group averages of E. As mentioned earlier in connection with Fig. 1, variability of average E values reduces as the group size increases from 3 to 12. Error bands ($\pm$ 10 and $\pm$ 20% about the population mean E value for the 9/99 material at 816°C) to determine the minimum number of tests required to estimate the average E value and the population mean E value (μ) are also indicated in Fig. 2. Three tests were sufficient to estimate the population mean of E to within 20%, however, at least four tests were required to estimate population mean to within 10% for the 9/99 material at 816°C. Similar analyses were conducted to determine the minimum number of required tests for E, PLS, ITS and SF for both the 9/99 (Table III) and the 1/01 (Table IV) materials at 816 and 1204°C. For brevity, only selected examples of the analyses are illustrated in the paper. Variations in the group averages for PLS (9/99 material at 816°C) are indicated in Fig. 3. Even though only three tests were required to estimate the population mean of PLS to within 20%, as in the case of E (Fig. 2), as many as eight tests (a significantly higher number) were necessary to estimate the population mean of PLS to within 10%. Figures 4 and 5 illustrate the variations in the group averages for ITS and SF, respectively, for 1/01 material at 1204°C. In the case of ITS, three and six tests were required to estimate the populations mean of ITS to within 20 and 10%, respectively. For SF of 1/01 material at 1204°C, seven tests were sufficient to estimate the population mean to within 20%. However, even 12 tests were not sufficient to estimate the population mean of SF to within 10%.

Table III. Minimum Number of Tensile Tests Required for MI SiC/SiC Material (9/99)

Temperature	Percent Error	E	PLS	ITS	SF
816°C	$\pm$10	4	8	6	9
1204°C	$\pm$10	4	11	4	12
816°C	$\pm$20	3	3	3	4
1204°C	$\pm$20	3	5	3	3

Table IV. Minimum Number of Tensile Tests Required for MI SiC/SiC Material (1/01)

Temperature	Percent Error	E	PLS	ITS	SF
816°C	$\pm$10	10	10	4	11
1204°C	$\pm$10	10	>12	6	>12
816°C	$\pm$20	3	5	3	4
1204°C	$\pm$20	4	5	3	7

DISCUSSION

Estimates of sample sizes based directly from the entire tensile database (all the 24 data points) might yield inappropriate results as they implicitly assume normally distributed parent populations for the different tensile properties. As mentioned before, the means of the randomly sampled groups always follow a normal distribution [5]. Therefore, the random sampling simulation with different group sizes used in this investigation is applicable for any type of parent population distribution (i.e., normal, log-normal, and Weibull etc.). In the case of 9/99 material at both 816 and 1204°C, five tests were sufficient to estimate the population mean values of all the four tensile properties to within 20%, whereas seven tests were needed for the 1/01 material (Tables III and IV). A total of 12 tests were able to predict the mean values of tensile properties to within 10% of the corresponding population means for the 9/99 material. However, in the case of the 1/01 material more than 12 tests would be needed to achieve similar estimates. Note that group sizes of more than 12 random samples were not investigated in this study. In general, the minimum number of tensile tests required to characterize the average values of all the four tensile properties for the 1/01 material are higher than those for the 9/99 material. This result from the random sampling and simulation of groups study is in agreement the observation made earlier regarding the standard deviations of the tensile properties of 1/01 material being larger than those for the 9/99 material (Tables I and II). Based on the results of both the 9/99 and 1/01 materials for the MI SiC/SiC composite, at least seven tests were required to estimate the mean values of the tensile properties of the material to within 20% of the average values of the populations. However, more than 12 tests would be required to estimate the mean values of the tensile properties to within 10%.

SUMMARY

Tensile properties (elastic modulus, proportional limit strength, in-plane tensile strength and strain to failure) of two variants (9/99 and 1/01) of woven, melt-infiltrated, SiC/SiC composite were characterized at 816 and 1204°C by conducting tensile tests. Average tensile properties obtained from both variants of the CMC were statistically compared to evaluate significant differences. At both temperatures, statistically significant differences were observed between the two variants of the CMC for the average in-plane tensile strength and strain to failure. Such significant differences were not observed for the average elastic modulus and proportional limit strength between the two variants. Simulation of groups by random sampling was performed to determine the minimum number of tests required to estimate the average values of all the four tensile properties to within 10 and 20% of the corresponding population averages for both the 9/99 and 1/01 materials. Results from the simulation were used to recommend the minimum number of tests required for characterizing the average tensile properties of the MI SiC/SiC composite.

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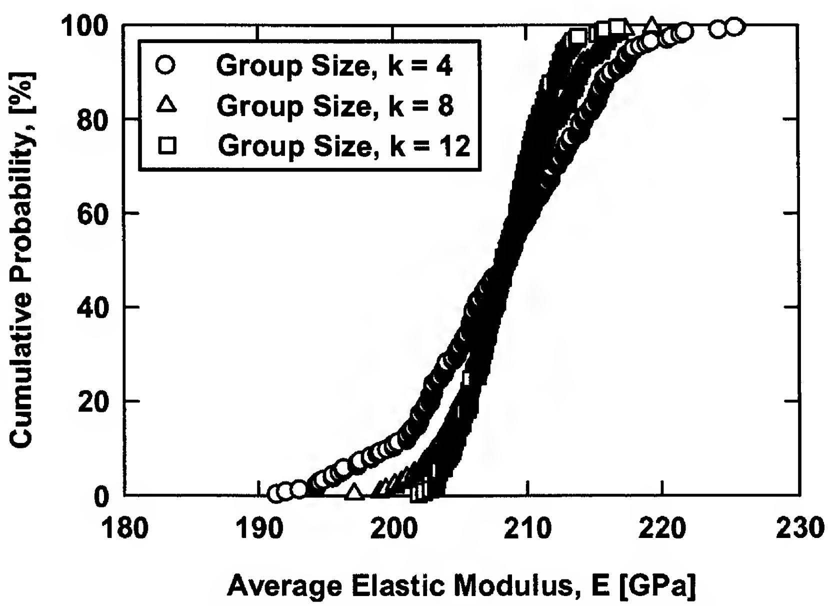

Figure 1: Distributions of the Group Averages for Elastic Modulus (9/99 Material at 816°C)

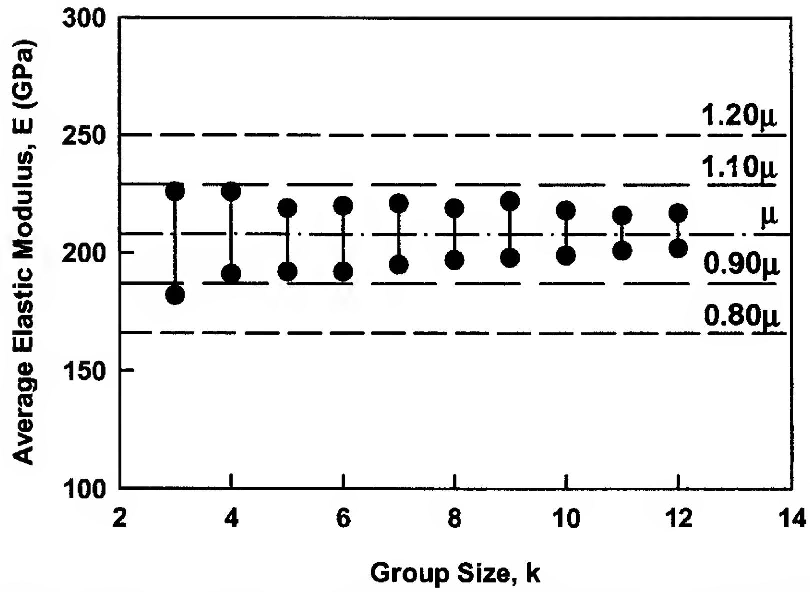

Figure 2: Variability in the Group Averages for Elastic Modulus (9/99 Material at 816°C)

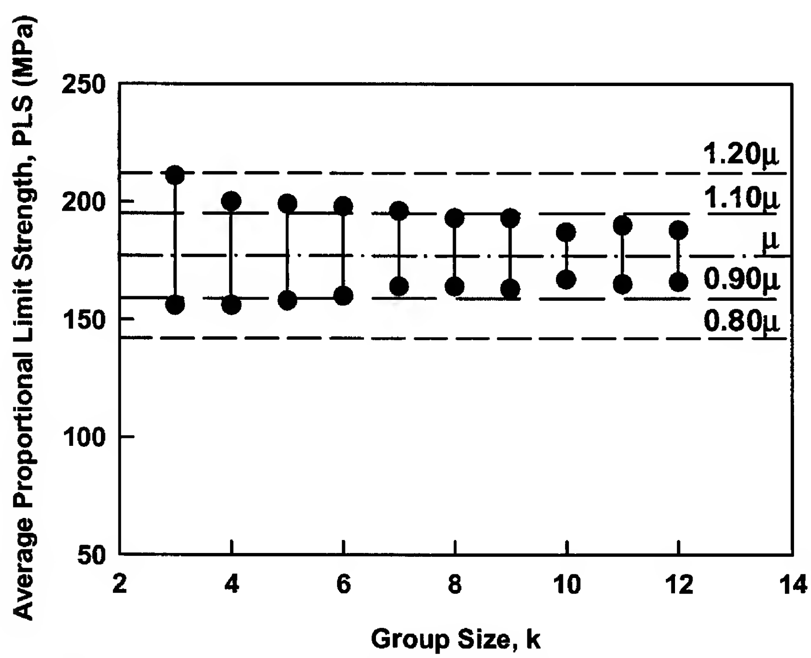

Figure 3: Variability in the Group Averages for Proportional Limit Strength (9/99 Material at 816°C)

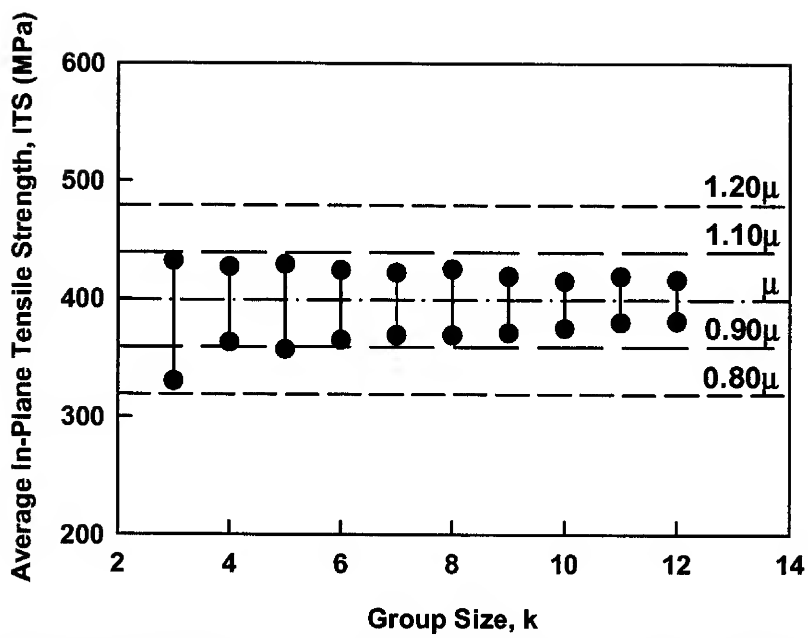

Figure 4: Variability in the Group Averages for In-Plane Tensile Strength
(1/01 Material at 1204°C)

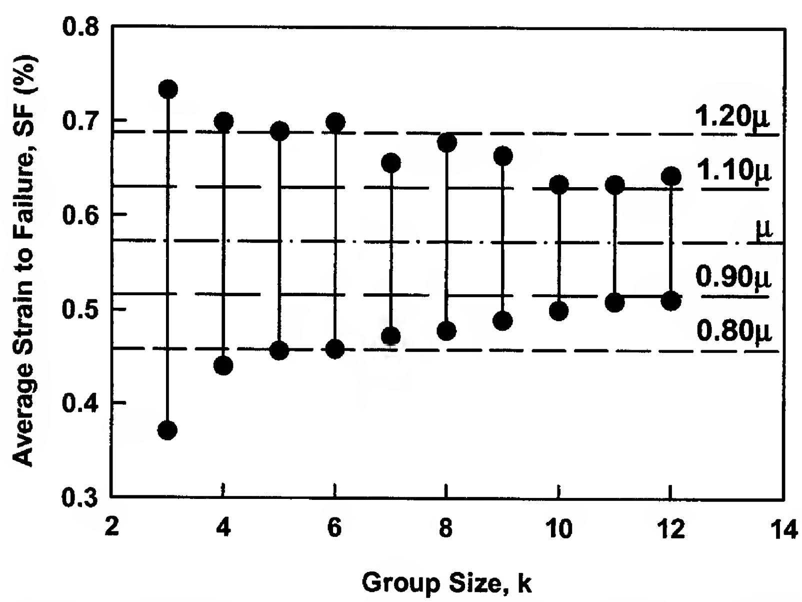

Figure 5: Variability in the Group Averages for Strain to Failure
(1/01 Material at 1204°C)